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Processing Technologies for Extracting Cobalt From Domestic Resources

By C. E. Jordan



UNITED STATES DEPARTMENT OF THE INTERIOR



Information Circular 9176

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

cm	centimeter	pct	percent
°C	degree Celsius	ppm	part per million
g/L	gram per liter	psi	pound per square inch
h	hour	st/d	short ton per day
lb	pound	μm	micrometer
lb/h	pound per hour	yr	year
mm Hg	millimeter of mercury		

PROCESSING TECHNOLOGIES FOR EXTRACTING COBALT FROM DOMESTIC RESOURCES

by C. E. Jordan¹

ABSTRACT

Domestic cobalt resources are relatively large, but low grade. The full potential for cobalt production from domestic sources is at least 19.4 million lb of cobalt per year exclusive of offshore resources. A summary of the cobalt processing technologies for the major domestic resources is presented in this Bureau of Mines report. The processing technologies for the Blackbird, Madison Mine, Duluth Gabbro, iron ore pyrite, laterites, and manganese sea nodules are nearly complete, but the economics are not favorable. Research on these resources should be limited to approaches that promise to cut the total processing costs by at least 50 pct. The most promising sources of cobalt are the spent copper leach solutions and siegenite from the Missouri lead ores. Research on cobalt processing from these two sources needs to be completed.

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INTRODUCTION

The United States is the largest consumer of cobalt in the world, but has no domestic cobalt production. Except for some scrap recycling, the United States depends entirely on foreign nations to supply over 15 million lb of cobalt annually. More than half of the cobalt metal comes from Zaire and Zambia (19).² With most of the cobalt coming from a small number of foreign producers, the United States is vulnerable to supply disruptions.

Cobalt is a critical element in many industrial and military products such as jet engine parts, high-strength tool steels, heat- and corrosion-resistant alloys, magnets, catalysts, drying additives in paints, and other chemicals. About two-thirds of the domestic cobalt consumption requires cobalt metal, either as a powder or as a high-purity cathode. The remaining third is as oxides or salts used in chemicals and paint drying additives (19). About half of the 15 million lb the United States consumes annually is considered essential (22). In recognition of the strategic importance of cobalt, research has been directed in three areas, recycling technology, cobalt

substitutes, and recovery technology from domestic resources. Currently, recycling accounts for about 8 pct of domestic consumption and has a potential for over 25 pct (19). Substitution technology has potential for an additional 25 pct, but research and development is moving slowly owing to the current low price of cobalt (22). Extensive research on primary cobalt production has been conducted on virtually every major cobalt-bearing deposit. In an effort to optimize future research and establish research priorities, the Bureau of Mines investigated the status of processing technologies for extracting cobalt from primary domestic resources.

Because domestic cobalt resources are low grade, cobalt production is likely only if metals such as nickel, copper, iron, lead, and zinc are recovered. There are two basic types of cobalt deposits, sulfide and oxide. The beneficiation and processing largely depends upon the type of ore and the associated metals. Each resource with over 15 million lb of Co is presented in this report (fig. 1). Along with the resource description, the beneficiation and processing technologies are presented with an assessment of the technical progress and the environmental problems.

²Underlined numbers in parentheses refer to items in the list of references at the end of this report.



FIGURE 1.—Domestic cobalt resources.

SULFIDE ORES

BLACKBIRD DEPOSIT

The Blackbird deposit near Salmon, ID, contains 0.65 pct Co with nearly all of the cobalt found in cobaltite (CoAsS) and a minor amount associated with chalcopyrite (CuFeS_2). The deposit's proven reserves are over 60 million lb of Co. Noranda Exploration Inc., Cobalt, ID is waiting for favorable economic conditions to build a beneficiation and cobalt processing plant with a planned capacity of 4 million lb of Co per year. The arsenic content of the ore has focussed some attention on the need for safe mining and tailings disposal. High cobalt and copper levels were found in streams draining from the deposit's historical mining site. An ion exchange process was successfully field tested to remove both the cobalt and copper from these runoff streams. The environmental costs associated with this deposit have been estimated at \$3.00/lb of Co produced.

Beneficiation of the Blackbird ore began with crushing and grinding the ore to 70 pct minus 200-mesh size. Using a sequential sulfide flotation process, chalcopyrite was floated first. Only 5 pct of Co was lost in the chalcopyrite concentrate containing 26 pct Cu and 0.65 pct Co. The remaining sulfides were floated, producing a bulk sulfide concentrate containing 5 pct Co, 0.1 pct Ni, and 0.4 pct Cu. A pilot plant was operated to demonstrate this process and 80 pct of the cobalt was recovered in the concentrate (10).

Historically, extraction of cobalt from the Blackbird concentrate began with a controlled oxidizing roast. However, because of the environmental problems associated with arsenic fumes, this technique is no longer considered appropriate. Fortunately, there are three hydrometallurgical alternative methods that do not require the oxidation roast. Cobalt can be leached as a sulfate, chloride, or ammine complex. Because a single company owns the Blackbird deposit, only the process proposed by Noranda Exploration Inc. will be

discussed here. The cobalt was dissolved by pressure leaching with sodium sulfate at 200° C and 150 psi oxygen pressure (fig. 2). Actually, the pyrite in the cobalt concentrate was oxidized, producing H_2SO_4 and $\text{Fe}_2(\text{SO}_4)_3$, which leached the cobalt from CoAsS . Sodium sulfate helped to suppress iron and arsenic dissolution in the autoclave. Ninety-seven percent of cobalt was extracted in a solution containing 30 g/L Co and 100 g/L H_2SO_4 . The leach slurry was cooled to 95° C and neutralized to pH 1.5, precipitating jarosite and ferric arsenate. The pregnant solution also contained nickel and other impurities such as iron, arsenic, copper, and zinc. A semicontinuous pilot plant using 22-lb batches was operated to demonstrate the extraction process. Commercial equipment for this high-pressure oxidation- H_2SO_4 leach process is available.

After extraction of the cobalt into solution, the refining process used largely depends upon the impurities and the final commercial cobalt product desired. Noranda chose to market a high-purity cathode cobalt. First, the hot pregnant solution was filtered from the leach residue, jarosite and ferric arsenate. At 50° C and 1.5 pH, H_2S was added to precipitate the copper and arsenic as sulfides. The filtered solution was oxidized and neutralized to pH 5.0 at 75° C to precipitate the residual iron. Zinc was removed from the filtered and cooled solution by ion exchange at pH 4.0 and ambient temperature. The solution pH was then adjusted to 2.5 and the nickel was removed by ion exchange. Finally, the cobalt was precipitated as cobaltous hydroxide with a lime slurry. After filtering the solution, the cobaltous hydroxide was leached with spent cobalt electrolyte followed by cobalt electrowinning at 50° C. The cobalt metal was stripped from the cathode and traces of hydrogen were removed by vacuum degassing at 820° C and 200 mm Hg. All of the unit operations, except the zinc and nickel ion exchange steps, are established commercial technologies.

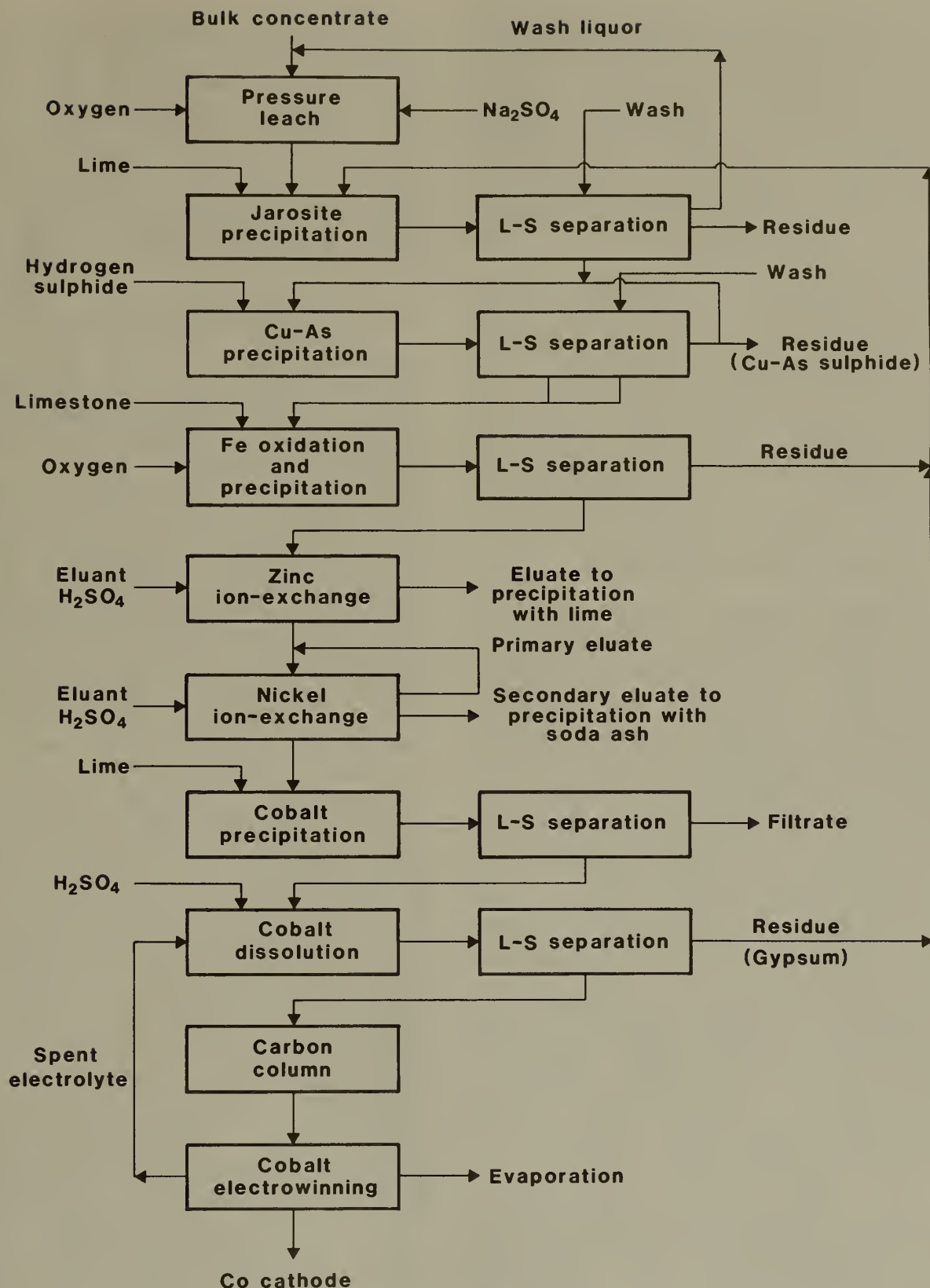


FIGURE 2.—Blackbird Mine cobalt process.

This refining process was also demonstrated in the semicontinuous miniplant previously mentioned. The overall cobalt recovery from ore to cathodes was 75 pct. The solution purification process removed the Cu, Zn, Ni, Fe, and As impurities and the cobaltous hydroxide precipitation removed soluble impurities such as Na and Mg. The cobalt cathode purity was excellent with the exception of a high selenium level (10).

The research on the Blackbird deposit is nearly complete. The extraction and refining technology has been demonstrated in a semicontinuous miniplant. The cobalt product met nearly all the required specifications and the environmental problems have been addressed. However, with estimated production costs at \$25/lb of Co, mining and processing this ore for cobalt was uneconomical.

DULUTH GABBRO DEPOSITS

The largest domestic Cu-Ni-Co sulfide resource is associated with the Duluth Gabbro Complex in northeastern Minnesota. Almost half of the cobalt is in cobalt-bearing sulfides that are disseminated throughout the deposit in the form of pyrrhotite, pentlandite, cubanite, and chalcopyrite. The remaining cobalt is associated with olivine and plagioclase. Over 184 million lb of Co is found in the deposit, which contains 0.025 pct Co, 0.18 pct Ni, and 0.79 pct Cu (24). Upon full development, the region could produce up to 5 million lb of Co per year.

Cobalt beneficiation of the Duluth Gabbro Complex ores has been limited to the sulfide minerals. The cobalt and nickel associated with oxides and silicates, about half of the deposit, has no beneficiation potential. The sulfide beneficiation began with crushing and grinding the ore to minus 200-mesh size. All the sulfides were floated with xanthate in a bulk concentrate. The xanthate collector was removed from the particle surfaces with lime. Then the concentrate was filtered and reground to minus 500-mesh size. The pentlandite and pyrrhotite were depressed and the chalcopyrite and cubanite were floated.

The bulk sulfide flotation process and the differential flotation process were demonstrated in a 12-st/d pilot plant. About 40 pct of the cobalt was recovered in the bulk sulfide concentrate. After the differential float, about 8 pct of the cobalt was in the copper concentrate containing 19 pct Cu, 0.5 pct Ni, and 0.05 pct Co. The remaining 32 pct of the cobalt was in the nickel-cobalt concentrate containing 0.5 pct Ni and 0.5 pct Co (27). The beneficiation technology for the Duluth Gabbro deposit appears to be complete. Differential flotation recovered 32 pct of the cobalt while increasing the grade from 0.025 to 0.5 pct. Environmental assessment of the mine tailings showed no potential environmental hazards.

The extraction of cobalt from the Duluth Gabbro is possible as a byproduct from copper and nickel recovery. Using the bulk sulfide concentrate, a matte smelting technique was used to produce a low-iron Cu-Ni-Co matte. The concentrate was roasted at 720° C to lower the sulfur content, fluxed with SiO₂ and CaO, and smelted at 1,300° C, making a Cu-Ni-Co matte. Only 25 pct of the concentrate cobalt was recovered in the matte which left most of the cobalt with the iron in the slag (28). From ore to matte, only 10 pct of the cobalt was recovered in the matte containing 61 pct Cu, 9 pct Ni, and only 0.1 pct Co. This was too high in copper and too low in Ni and Co for the world's commercial Cu-Ni refineries, which usually treat mixed sulfides or mattes containing more Ni than Cu. The cobalt was not recovered efficiently. A survey of six commercial Co-Ni-Cu matte smelting operations showed that between 30 to 60 pct of the cobalt was recovered in the matte. This indicates that matte smelting is not appropriate for efficient cobalt recovery. Alternative hydrometallurgical techniques need to be studied to establish cobalt recovery technology for the Duluth Gabbro concentrates. Afterwards, continuous testing up to pilot plant scale is needed for economic evaluation.

With half of the cobalt left in the flotation tailings, both the ore or the tailings may be suitable for solution

mining or heap leaching with weak acid. Exploratory test work should be conducted to see if the cobalt and other metals can be efficiently leached. The technology for recovering cobalt from similar leach solutions has been developed and will be discussed later in this report ("Spent Copper Leach Solutions" section). Leaching the ore or the tailings may result in lower costs in recovering cobalt from this resource.

YAKOBI ISLAND DEPOSIT

Another large Cu-Ni-Co deposit is found on Yakobi Island in southeastern Alaska. A resource of approximately 14 million lb of Co is estimated for this deposit. Most of the cobalt is associated with sulfides. The deposit also contains 85 million lb of Cu and 140 million lb of Ni (16). The average cobalt grade is 0.04 pct. The deposit has been explored, but very little beneficiation and extraction research has been conducted. The deposit is located in the National Wilderness Preservation System where future exploratory mining is restricted.

MADISON MINE

Cobalt and nickel mineralization is associated with lead deposits in southeastern Missouri. The Madison mine located on the edge of the lead belt has a high-grade cobalt ore, making it uniquely different than the other lead-zinc deposits. At the Madison Mine up to 21 million lb of Co is found in ore containing 0.17 pct Co. Siegenite, $(\text{Ni},\text{Co})_3\text{S}_4$, is the major cobalt mineral, but some cobalt is also found in chalcopyrite, sphalerite, galena, and millerite (32). The expected annual cobalt production by Anshutz Mining Co. was between 1 and 2 million lb (22), however, these plans are contingent upon more favorable cobalt prices.

After crushing and grinding, bulk sulfide flotation was used to beneficiate the ore. The method was demonstrated by processing 225 short tons of ore through a pilot plant. The cobalt concentrate ranged in grade from 1.5 to 3.9 pct Co and recoveries ranged from 95 to 87 pct,

respectively (9). Some research was conducted to sequentially float a Pb, Cu, and Co-Ni concentrate, but this technique could only be used with the high-grade portions of the ore. Beneficiation research for the Madison ore deposit appears to be complete. The technology has been tested on a pilot scale and no problems have been reported.

The cobalt was extracted from the bulk sulfide concentrate with a sulfation roast followed by pressure leaching with H_2SO_4 (fig. 3). A continuous pressure leaching circuit was operated and extracted 96, 94, and 87 pct of the Co, Cu, and Ni. Minor amounts of Fe, Mn, and Zn impurities were also extracted. Nearly all of the lead remained in the leach residue.

The copper in the pregnant solution was recovered by electrowinning. The spent copper electrolyte was purified with ammonia and NaHS to remove the iron, residual copper, manganese, and zinc. Solvent extraction of the purified solution using versatic acid recovered both cobalt and nickel. The organic was stripped into a chloride system, where another solvent extraction using tri-n-octylamine (TNOA) separated the cobalt from the nickel. The cobalt was stripped from the organic and electrowon, producing cobalt metal and chlorine gas. The chlorine was reacted with hydrogen to produce hydrochloric acid, which was recycled back to the cobalt-nickel stripping operation. The nickel raffinate was treated to precipitate marketable nickel salts. This refining method is currently practiced by the Sumitomo Metals Mining Co., LTD, Tokyo, Japan for the treatment of mixed sulfides (22). The operating costs for the Madison Mine are reported to be around \$20/lb of Co produced.

MISSOURI LEAD BELT DEPOSITS

The typical Missouri lead belt ore contains 5.9 pct Pb, 1.1 pct Zn, 0.3 pct Cu, 0.02 pct Ni, and 0.015 pct Co, which is present as siegenite. These ores are a primary source of lead, zinc, and some copper. Based upon the annual production from Missouri lead belt mines, 2.5 million lb of Co is mined, but not

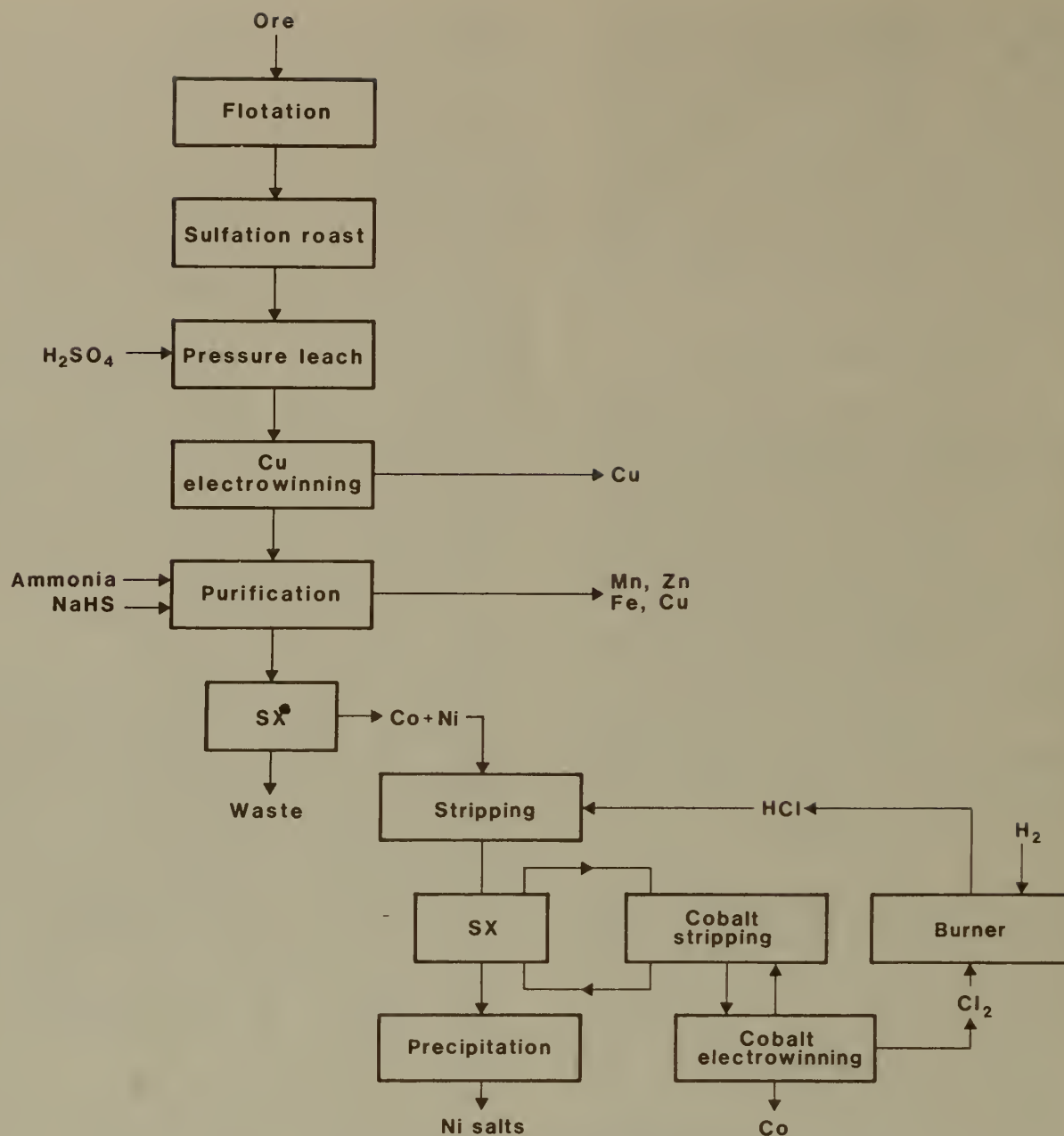


FIGURE 3.—Madison Mine cobalt process.

recovered. About 80 million lb of Co is present in the unmined ores.

With beneficiation circuits already in place to recover lead, zinc, and sometimes copper, research on the recovery of cobalt has been aimed at the mineral concentrates and the tailings product. About 20 to 30 pct of the cobalt ends up in the chalcopyrite concentrate containing 29 pct Cu, 0.9 pct Co, 1.2 pct Ni, 6 pct Pb, and 0.5 pct Zn. The siegenite was finely interlocked with the

chalcopyrite, which needed to be ground below 10- μ m size to obtain liberation. After grinding, the chalcopyrite was refloats in several stages leaving the siegenite behind. The process was demonstrated on a 130-lb/h bleed stream from a lead mill copper circuit. About 80 pct of the Co was recovered in a siegenite concentrate containing from 2.8 to 3.8 pct Co and from 4 to 5 pct Ni.

About half of the cobalt in the Missouri lead ores ends up in the flotation

tailings, which contain 0.02 pct Co. Flotation with a mixture of amine and xanthate recovered 66 pct of this Co in a bulk sulfide concentrate containing 0.11 pct Co. The process is currently being pilot tested at Cominco American, Inc's Magmont Mine, Iron County, Missouri.

The remaining 20 to 30 pct of the cobalt is in the galena and sphalerite concentrates (23). Here the siegenite is finely interlocked with both the galena and sphalerite. Physical separation of the siegenite from these sources has not been tested. The beneficiation research for the Missouri lead ores appears to be nearing completion. About 20 pct of the ore's cobalt is recovered in the siegenite concentrate from the copper circuit. Bulk sulfide flotation has recovered 33 pct of the ore's cobalt from the flotation tailings. However, this bulk sulfide concentrate is low grade. The potential for increasing this grade and maintaining cobalt recovery appears to be low, because the siegenite is interlocked with the other sulfide minerals at grain sizes below 5 μm (8).

Recovery of cobalt from the siegenite concentrate appears to be linked to marketing the concentrate to a cobalt refinery. The nickel content of this concentrate was lower than specifications for the world's Co-Ni-Cu refineries (22). However, it is possible that this concentrate could be blended with higher grade nickel concentrates for cobalt extraction. The next likely processing route is the method proposed by Anshutz Mining Corp., Fredricktown, MO, for the Madison Mine. This method should be tested on the siegenite concentrate to determine its feasibility and to obtain a preliminary economic evaluation of the overall cobalt recovery process.

No technology has been developed for the low grade, bulk sulfide concentrate from the lead mill tailings. The dolomite gangue will probably exclude acid leaching leaving ammoniacal leaching as the only potential extraction technology for this concentrate. Research needs to be conducted to determine the technical feasibility of cobalt extraction from this concentrate.

COPPER DEPOSITS

Some of the primary copper ores of Utah, Arizona, and New Mexico contain cobalt at levels around 0.01 pct Co. A specific cobalt mineral has not been identified, but the cobalt appears to be more concentrated in the iron sulfide minerals. Although less than 10 pct of these deposits have significant concentrations of cobalt, with over 12 billion st of ore in reserves, these deposits still could contain over 75 million lb of Co (32). Possible by-product recovery of the cobalt has been identified in two portions of the copper production circuits. Pyrite and pyrrhotite in some flotation tailings contain 0.06 pct Co and spent copper leach solutions from some heap leaching operations contain 15 to 30 ppm Co. At least two deposits have been identified containing cobalt-bearing sulfides with a potential for producing 1.0 million lb of Co annually. Five leach solution sources have been identified with a potential for producing over 1.5 million lb of Co annually.

Pyrite Concentrates

Flotation of pyrite from the tailings of one Arizona mine produced a sulfide concentrate containing 0.06 pct Co. About 30 pct of the cobalt was recovered along with 50 pct of the copper. No research has been done to extract cobalt from this low-grade cobalt concentrate. Beneficiation and extraction methods need to be developed and preliminary economics of a process should be evaluated. The pyrite concentrates have nearly the same grade as most of the domestic laterites, but do not have the mining costs.

Spent Copper Leach Solutions

The second cobalt source from primary copper operations is the spent copper leach solutions. Heap leaching operations extract the cobalt from the ore into a weak acid solution. The degree of extraction is not known. However, after recovery of the copper, the spent copper leach solution contained 15 to 30 ppm Co

(17). Ion exchange was used to recover the Co, Ni, and Cu from this solution (fig. 4). The loaded resin was sequentially stripped with (1) weak H_2SO_4 to remove a portion of the Fe, Zn, and Al; (2) 30 g/L H_2SO_4 to recover the cobalt and nickel; and (3) NH_4OH to remove the copper and residual nickel. Next, solvent extraction using di(2-ethylhexyl) phosphoric acid was used on the cobalt-nickel solution to remove the Fe, Zn, and Al impurities. Solvent extraction with

CYANEX 272³ was used on the cobalt-nickel bearing raffinate to recover the cobalt. The organic phase was stripped with spent cobalt electrolyte, followed by cobalt electrowinning. The process was demonstrated continuously with a multiple-compartment ion exchange unit and recovered 96 pct of the Co from the spent

³Reference to specific products does not imply endorsement by the Bureau of Mines.

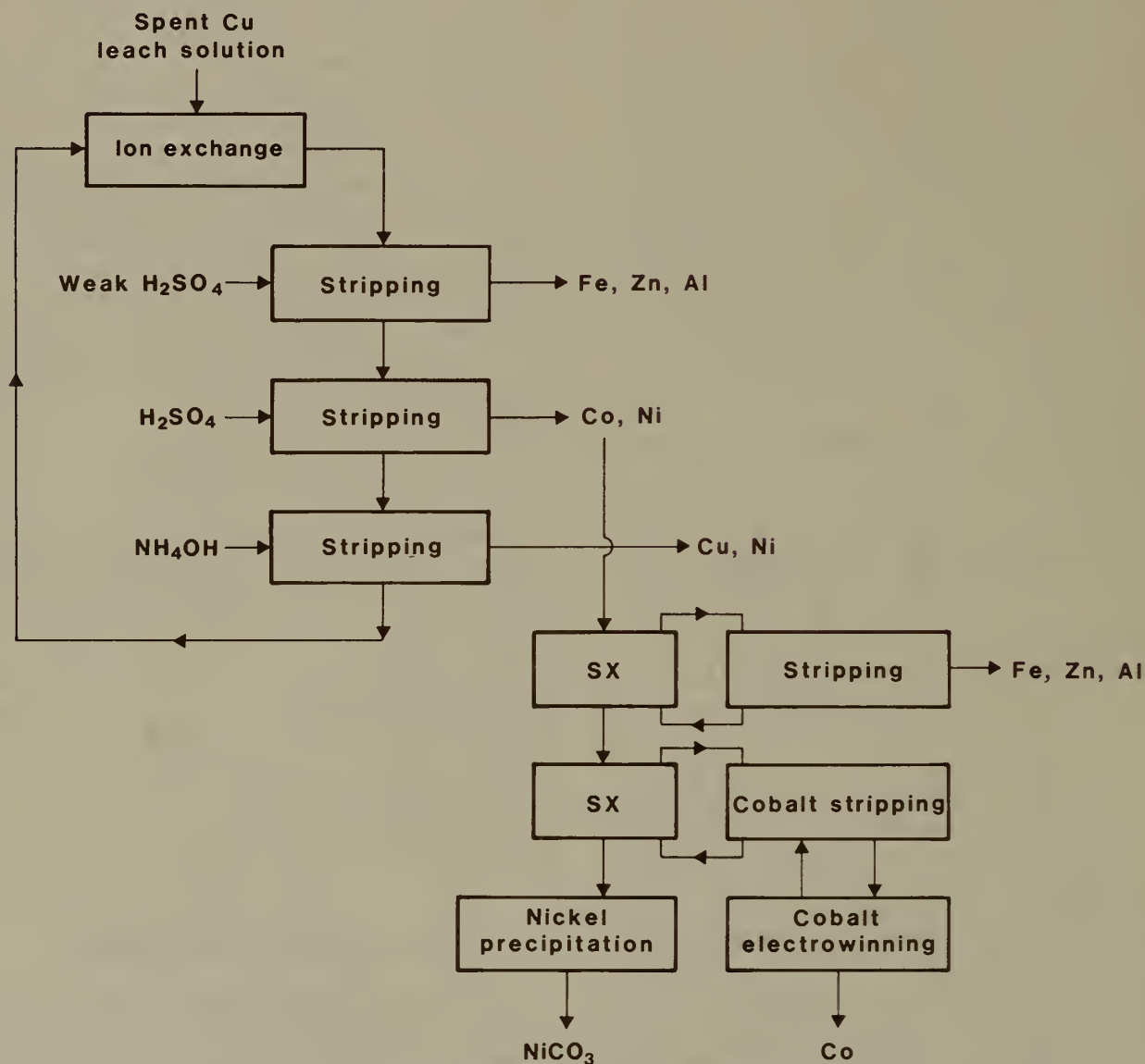


FIGURE 4.—Cobalt from spent Cu leach solution.

copper leach solution (18). An economic evaluation of the process from spent copper solution to cathode cobalt metal showed that, with credits for the copper and nickel values, cobalt could be produced for about \$7.00/lb.

The research on cobalt recovery from spent copper leach solutions is nearly complete. A long-term pilot program may be needed to establish resin life and the steady-state solution concentrations. An economic evaluation indicated that this cobalt source has the highest potential for domestic production.

IRON DEPOSITS

Cobalt is also found in some iron deposits, associated with sulfides. In the iron deposits of Cornwall, PA, 56 million lb of Co is present at grades ranging from 0.02 to 0.056 pct Co. About 40 to 60 pct of the ore is magnetite and 3.5 pct pyrite (29). Historical processing of these ores recovered byproduct Cu, Co, Ag, and Au. In the 2 yr prior to closure of these iron mines, the cobalt grade dropped by 30 pct. However, it was not clear from that report whether the grade of cobalt in the sulfides dropped or if the sulfide content of the ore dropped. Before the iron mines closed, 1.5 million lb of Co was produced annually.

Beneficiation of these ores began with crushing and grinding followed by magnetic separation to recover a high-grade magnetite concentrate (fig. 5). The non-magnetic product was floated to recover a bulk sulfide concentrate, mostly pyrite, containing 0.7 to 1.4 pct Co.

Cobalt extraction from the pyrite concentrate began with roasting and shipping the calcine product to Pyrites Co. Inc. in Wilmington, DE. The calcine was percolation leached with sulfuric acid for an average of 250 h. The Co, along with some of the Fe, Cu, and Mn, was dissolved into the solution. The extraction percentage was not reported.

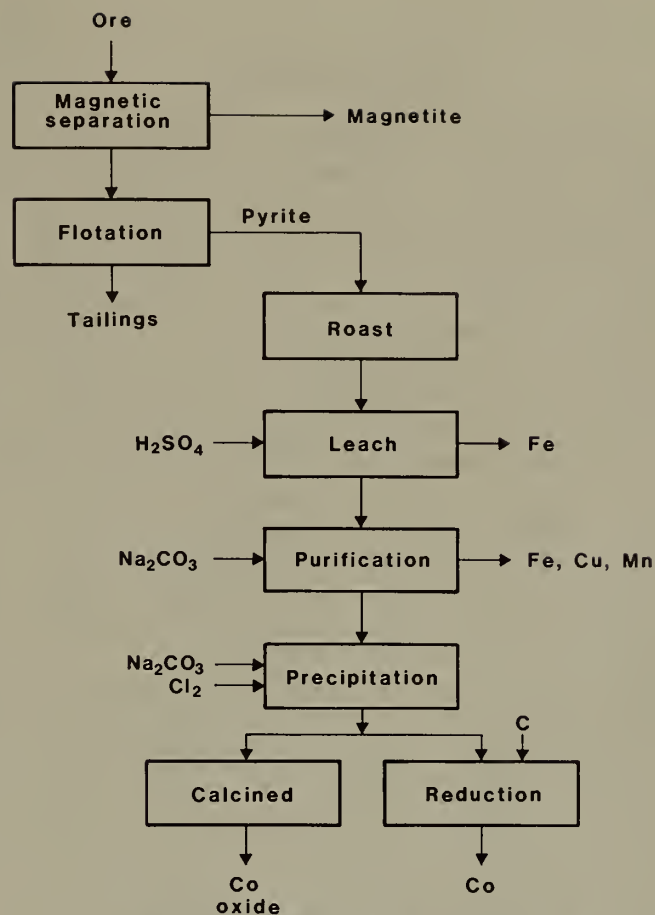


FIGURE 5.—Cobalt from Pennsylvania iron ore.

The pregnant solution was purified with sodium carbonate, which precipitated the Fe, Cu, and Mn. After filtering, chlorine and more sodium carbonate were used to precipitate the cobalt. The filtered cobalt cake was either calcined to the oxide containing 70 pct Co or reduced to the Co metal with charcoal. The final metal powder contained 98 to 99 pct Co. Over 99 pct of the cobalt in the solution was recovered (33). This process was used commercially up until 1971 when the mining companies closed their iron mines, cutting off the supply of byproduct cobalt-bearing pyrite. If iron ore markets improve, this source of cobalt may resume production.

OXIDES

LATERITE DEPOSITS

The laterite deposits of northwestern California and southwestern Oregon are the result of extensive weathering of ultramafic serpentine rocks and contain over 96 million lb of Co. However, the deposits are located in a wilderness preserve. The average grade is 0.08 pct Co, but varies throughout the deposits from 0.06 to 0.25 pct Co. There is also 0.5 to 1.2 pct Ni, 2 pct Cr, and 0.3 pct Mn in these deposits. Nearly all of the Co is found in the manganese mineral, which makes up only 1 pct of the ore. Well over a third of the ore is goethite, which contains most of the nickel. The remainder of the ore is quartz, hematite, magnetite, and chromite (5).

Ore beneficiation of the laterite deposits has been limited to crushing and screening out some of the coarse pieces of quartz (30). The grain size of the goethite and manganese mineral is very small, which would require fine grinding of the ore to obtain liberation. No successful beneficiation techniques have been developed for this fine-grain mineral separation. Even with a perfect separation, the Co and Ni grades would only be 2.5 times larger than in the ore. Considering the limited beneficiation potential and the lack of technology, these laterite ores will not be significantly beneficiated before the extraction process.

The extraction of cobalt from laterites has received considerable research attention. The two major techniques for cobalt extraction are reduction roast-ammoniacal leach (fig. 6) and H_2SO_4 leach (fig. 7). The basic reduction roast-ammoniacal leach process shown in figure 6 began with drying and crushing the ore to minus 2-cm size. Selective reduction of the nickel and cobalt with H_2 and CO was conducted in multiple hearth furnaces at 700° to 760° C. The reduced ore was leached with an aerated solution of ammonium hydroxide and ammonium carbonate. The nickel and cobalt were leached out while the iron remained insoluble. This process, commonly called the Caron

process, was used in Australia and the Philippines. Only about 50 pct of the cobalt and 75 to 80 pct of the nickel were recovered.

The Bureau's reduction roast-ammoniacal leach process was tested on laterite ores (30). Crushed pyrite was added to the ore followed by multiple-hearth roasting at 525° C with pure CO gas. The nickel and iron combined to form ferronickel and the copper and cobalt were reduced to the individual metals. After cooling, the calcine was finely ground and leached with ammonium hydroxide, ammonium sulfate, and oxygen to dissolve Co and Ni as ammine complexes. The process was demonstrated in a 500 lb/d integrated, continuous operation using an ore sample containing 0.2 pct Co and 0.97 pct Ni. About 85 pct of the Co and 90 pct of the Ni were recovered in the pregnant leach solution. However, with ore samples containing 0.05 to 0.1 pct Co (average, 0.08 pct), a 4.5-st/d pilot plant test of this process only extracted 67 pct of the Co and 83 pct of the Ni (31).

Laterites were also processed with a H_2SO_4 leach process (fig. 7) (3). The ore containing 0.08 pct Co and 1 pct Ni was slurried and pumped to leaching towers where it was leached with H_2SO_4 at 200° to 250° C under more than 500-psi pressure. The process dissolved cobalt, nickel, and magnesium, while the iron was hydrolyzed and precipitated. In laboratory tests, about 85 to 90 pct of the cobalt and 90 to 95 pct of the nickel was extracted. California Nickel Corp., San Francisco, CA completed pilot-plant work on this process and AMAX proposed to employ this process for the New Caledonia COFREMI laterite operations to recover nickel and cobalt (2). To minimize acid consumption, the process is generally restricted to low-magnesia ores (less than 5 pct MgO). The California and Oregon laterites contain 4.3 to 7.5 pct MgO as serpentine and chlorite.

The solution refining process depends upon the extraction method. For the ammoniacal solutions, $NH_4H_2PO_4$ was added to precipitate Mg and Mn as (Mg, Mn) NH_4PO_4 , a fertilizer byproduct. After

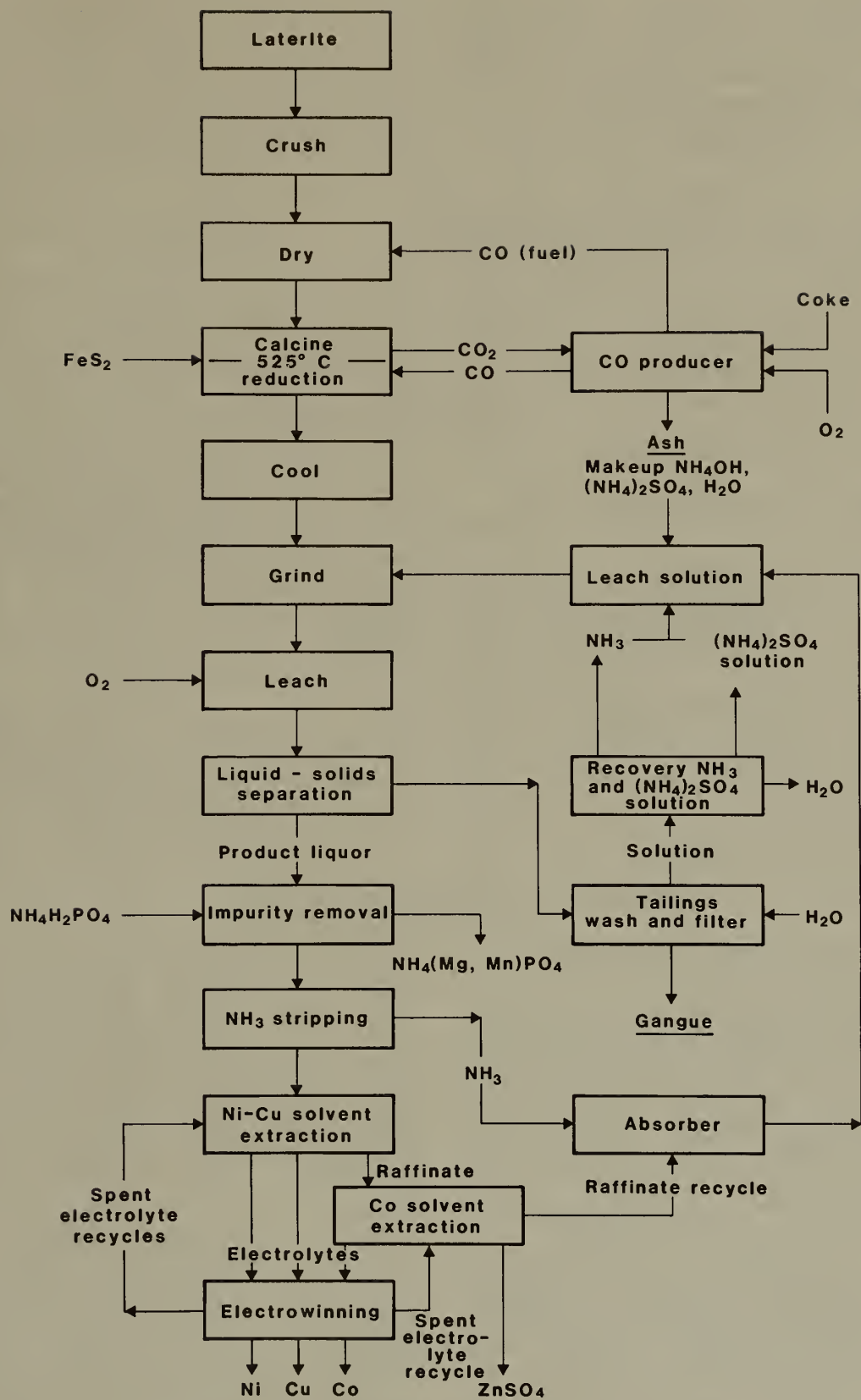


FIGURE 6.—Reduction roast-ammoniacal leach process for laterites.

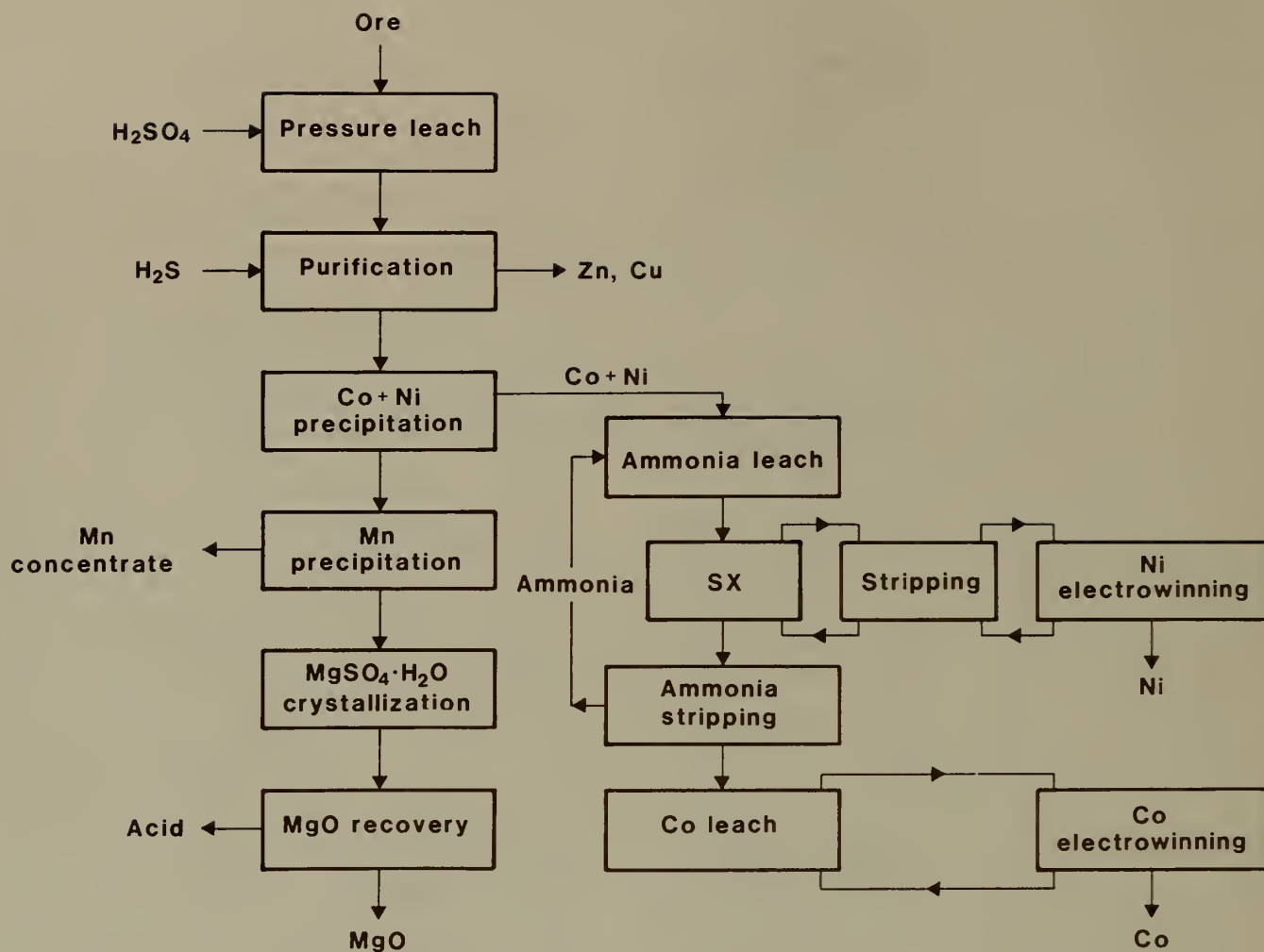


FIGURE 7.—Sulfuric acid leach process for laterites.

stripping excess ammonia from the solution with steam, solvent extraction was used to remove both the Ni and the Cu. The nickel was stripped from the organic with spent nickel electrolyte followed by a electrowinning the nickel. The copper was then stripped from the organic with spent copper electrolyte followed by copper electrowinning. The Ni-Cu raffinate was neutralized precipitating the residual Cu, Mg, Zn, Mn, and Ni. The cobalt in the solution was reduced from Co^{3+} to Co^{2+} with cobalt shot and solvent extraction used to recover the cobalt. The organic was stripped with spent cobalt electrolyte and the cobalt was electrowon. This process was demonstrated in a pilot plant. From ore to metal only 65 pct of the cobalt and

83 pct of the nickel were recovered in the cathode products (31).

For the H_2SO_4 extraction refining process, H_2S was added to precipitate Cu and Zn as sulfides. Next the Ni and Co were precipitated with lime. Then the Ni-Co precipitate was leached with ammonia solution, followed by solvent extraction. The organic phase was stripped with spent nickel electrolyte and the nickel was electrowon. The raffinate was treated with steam to strip the ammonia and then the cobalt was precipitated. The cobalt precipitate was leached with spent cobalt electrolyte followed by cobalt electrowinning. A 5-st/d pilot plant was operated to demonstrate this technique. From ore to metal over 90 pct of the Co and Ni were

recovered (2). Research on laterite processing appears to be complete, however, the economics are not favorable. The residues have been evaluated and proved to be nontoxic to the environment (25), but development of these deposits will face significant opposition from environmentalists.

MANGANESE SEA NODULES

Manganese nodules are found over large areas of the ocean floor at depths of around 2,000 to 18,000 ft. They range from 0.25 to 3 in. in size and contain 25 to 30 pct Mn, 1.0 to 1.5 pct Ni, 0.5 to 1.0 pct Cu, and 0.25 pct Co (26). Large concentrations are found in the east central Pacific area. Only a small portion of nodule samples were taken from the U.S. Exclusive Economic Zone (EEZ) located off the Hawaiian shore and other islands of the Trust Territory of the Pacific Islands and affiliated territories. Estimates as high as 1 billion lb of Co have been made for this resource.

The mineralogy is basically fine grained oxides mixed with layers of silicious gangue minerals (13). Although a number of problems remain to be solved, the mining technology proposed for deep-sea nodules has been a hydraulic air suction system or a continuous-line bucket system. Much of the prototype equipment has already been designed, patented, built, and tested (15). However, international politics and many legal problems appear to have stalled the development of this resource.

The fine grain mineral structure of the nodules precludes any effective beneficiation except for physically separating the nodules from the bottom sediment by screening.

Extraction Methods

A great deal of research has been conducted to develop methods for extracting the Mn, Cu, Ni, and Co from the nodules (12). Five of the most promising techniques for cobalt extraction are (1) gas reduction and ammoniacal leach,

(2) cuprion ammoniacal leach, (3) high-temperature and high-pressure H_2SO_4 leach, (4) reduction and HCl leach, and (5) smelting and H_2SO_4 leach.

Gas Reduction and Ammoniacal Leach Process

First, the nodules were ground to 65-mesh size and dried followed by high-temperature ($625^\circ C$) reduction of manganese dioxide to manganese oxide by CO gas (fig. 8). After cooling to $40^\circ C$, the Cu, Ni, and Co were dissolved with an oxidizing ammoniacal ammonium carbonate leach (Caron process). About 90 pct of the Cu, 90 pct of the Ni, and 70 pct of the Co were dissolved into the solution.

Cuprion Ammoniacal Leach Process

The nodules were ground to 65-mesh size and reduced with cuprous ions (Cu^+) in a ammoniacal solution at $50^\circ C$ (fig. 9). The manganese dioxide was reduced to manganese oxide. The cobalt was leached with a strong solution of ammonia and carbon monoxide. Next, the slurry was oxidized with air to oxidize the soluble ions and precipitate the iron. Only 50 pct of the Co was recovered, but 90 pct of the Cu and Ni was recovered (1). This extraction technique is not appropriate for cobalt recovery.

High-Temperature, High-Pressure H_2SO_4 Leach Process

The nodules were ground to 65-mesh size and leached with H_2SO_4 at $245^\circ C$ and 500-psi pressure (fig. 10). The recovery of Co, Cu, and Ni was 90, 95, and 95 pct, respectively. Small amounts of Fe, Mn, and Zn were also dissolved.

Reduction and HCl Leach Process

The nodules were ground to 65-mesh size and dried, followed by high-temperature ($500^\circ C$) gaseous HCl reduction of manganese dioxide to manganese chloride (fig. 11). This reaction also produced chlorine gas that reacted with the other metal oxides, forming metal chloride salts.

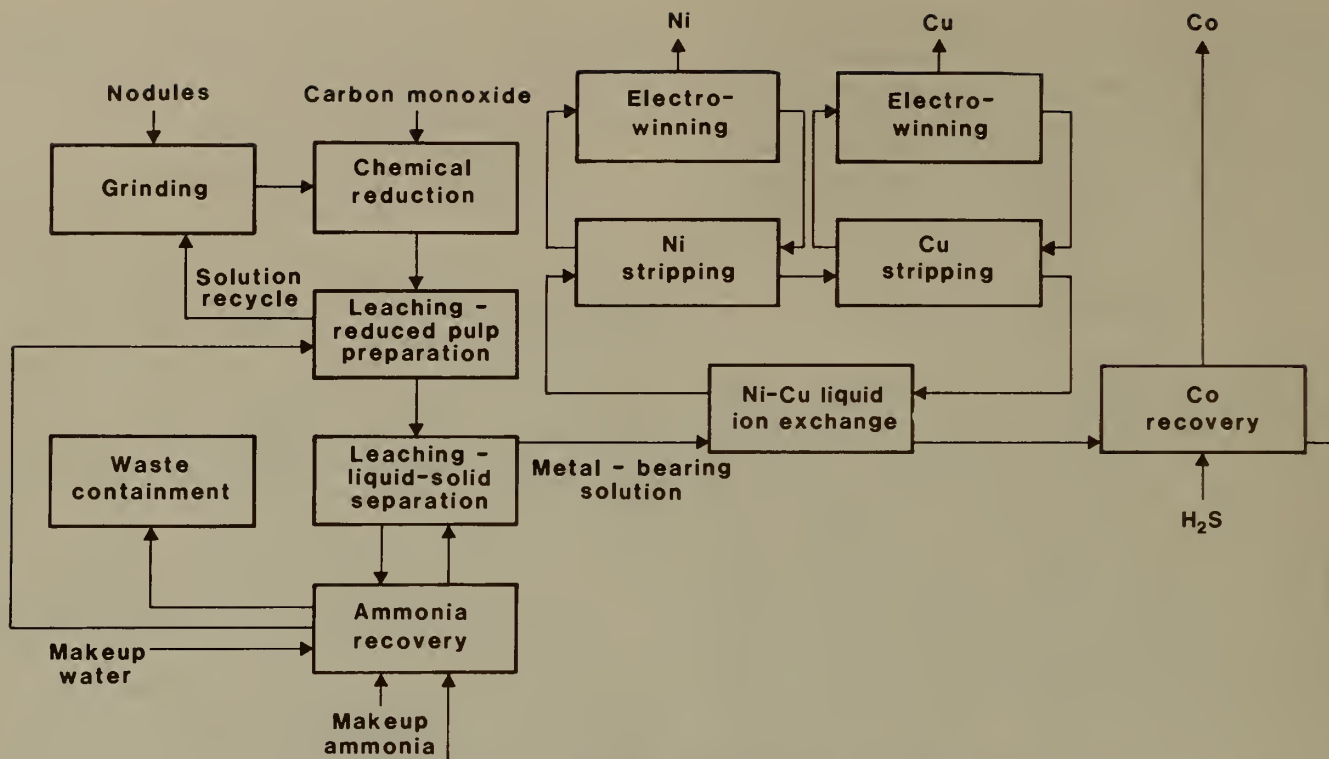


FIGURE 8.—Gas reduction and ammoniacal leach process for sea nodules.

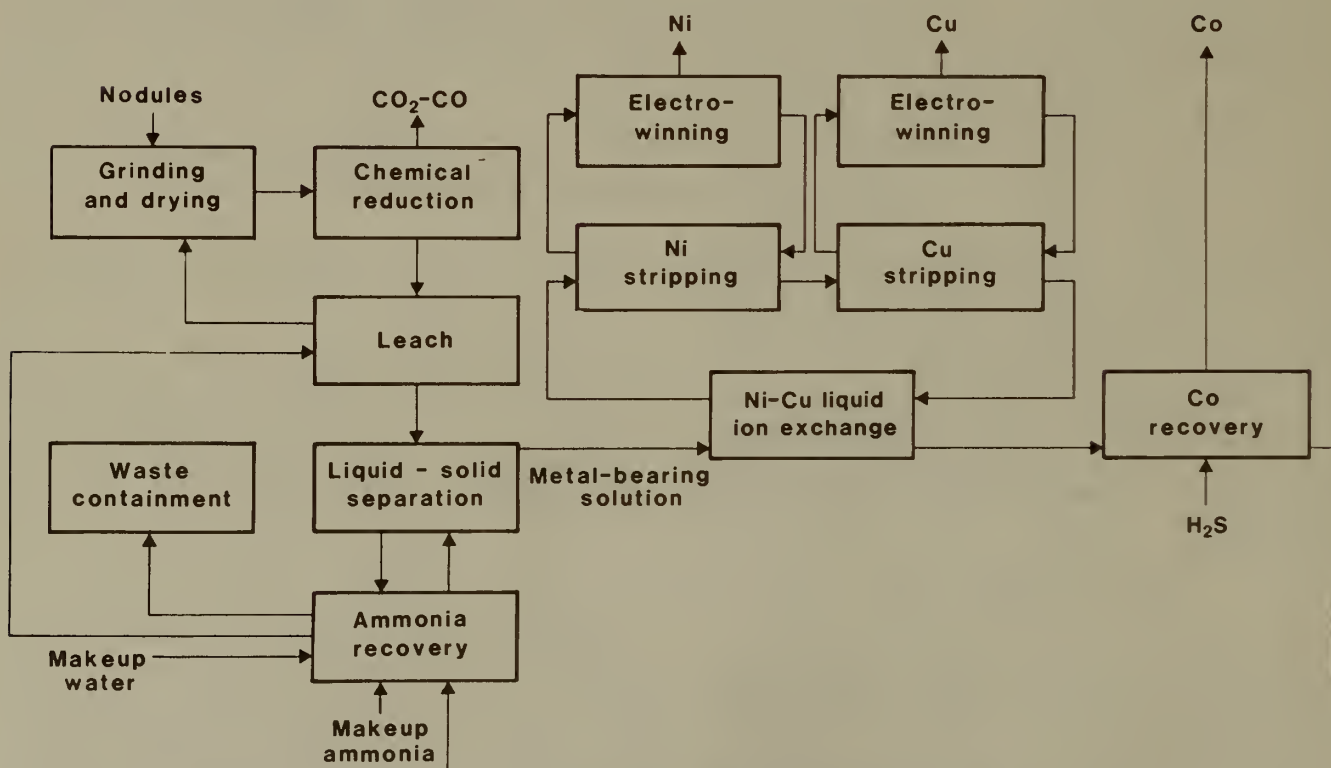


FIGURE 9.—Cuprion ammoniacal leach process for sea nodules.

Smelting and H_2SO_4 Leach Process

The nodules were dried, roasted with coke and CO gas at 725°C , and smelted with silica flux at $1,425^\circ\text{C}$ (fig. 12). The slag, containing Mn, Fe, and SiO_2 , was removed and subsequently used for ferromanganese production. The manganese alloy containing the Fe, Cu, Ni, and Co was converted by adding sulfur and heating to form a matte and an iron alloy. More silica flux was added and the matte-iron alloy was blown with air to lower the iron content of the matte to 5 pct. The finished matte was granulated, ground to 325-mesh size, and pressure leached with H_2SO_4 at 150 psi and 110°C . About 90 pct of the Co was dissolved in the solution along with 90 pct of the Cu and Ni. The Co recovery was uncommonly high for a matte smelting process.

Refining Process

Refining of the leach solutions depends upon the solution type such as ammoniacal, acid chloride, or acid sulfate.

Ammoniacal Solution Refining

For the gas reduction and ammoniacal leach process and cuprion process, the pregnant liquor passed through a solvent extraction circuit where the copper, nickel, and some ammonia were removed. Most of the ammonia was removed from the organic phase by washing with a weak aqueous ammonia solution. The organic phase was cleansed of the residual ammonia with a slightly acidic ammonium sulfate solution. The nickel was carefully stripped from the organic phase using spent nickel electrolyte containing

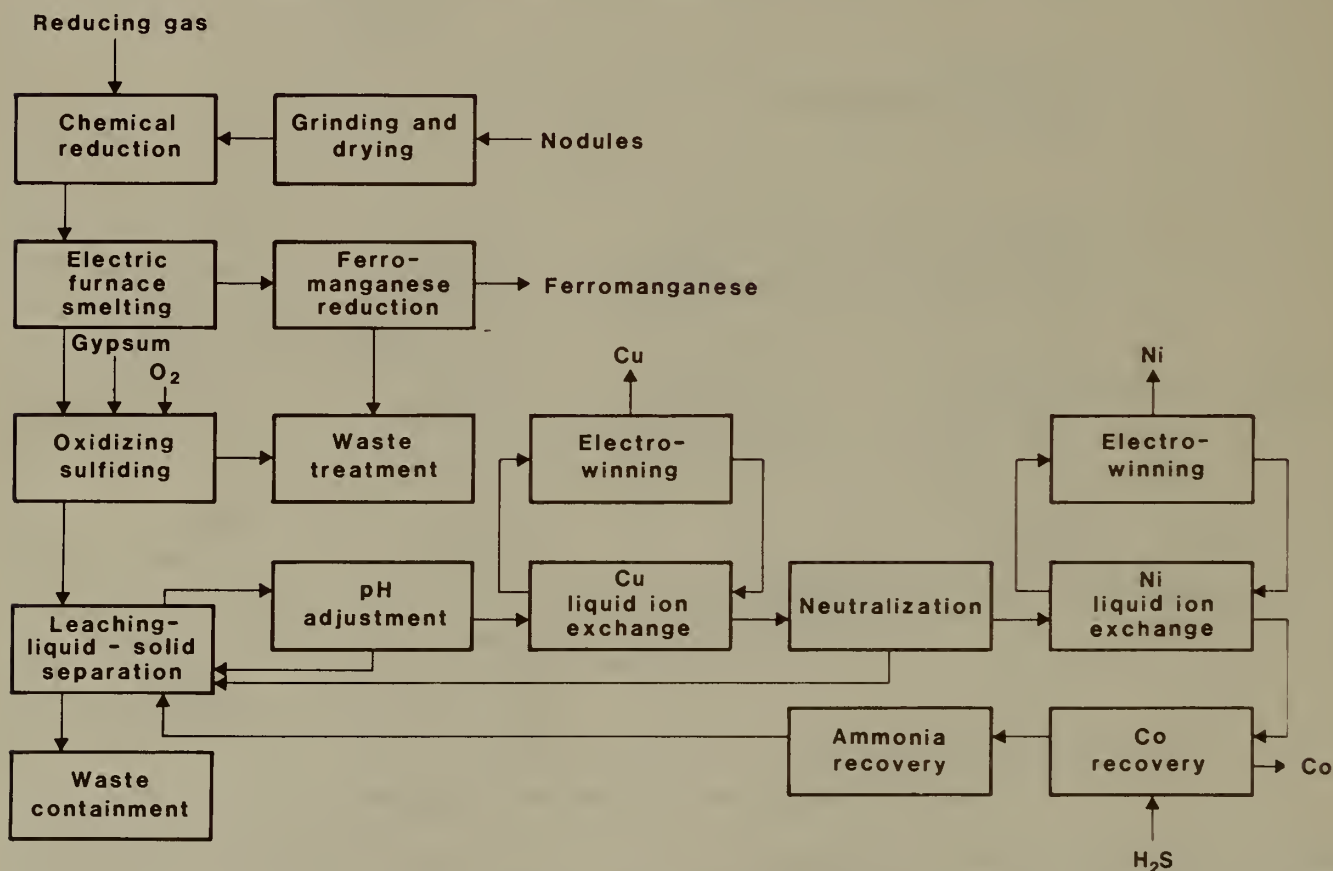


FIGURE 12.—Smelting and H_2SO_4 leach process for sea nodules.

40 g/L H_2SO_4 . Then the nickel was electrowon from the nickel-rich electrolyte. The copper was stripped from the nickel-free organic phase using a spent copper electrolyte containing 160 g/L H_2SO_4 . The copper was electrowon from the copper-rich electrolyte and the metal-free organic was recycled to the solvent extraction circuit.

The LIX raffinate was treated with ammonium hydrosulfide, which precipitated the Co along with residual Cu, Ni, and Zn. The sulfide mixture was pressure leached with air to preferentially dissolve the Ni and Co, leaving the Cu and Zn sulfides behind in the residue. After filtering off the residue, hydrogen sulfide was used to remove the residual Zn and Cu impurities. Then the solution was heated in an autoclave with hydrogen to reduce the Ni. The nickel powder was washed, dried, and briquetted. The cobalt sulfate solution was concentrated in an evaporator-crystallizer to precipitate the Co and residual Ni with ammonium sulfate. The salts were redissolved with a strong ammonia solution and oxidized with air, converting the Co^{2+} to Co^{3+} . The remaining nickel was precipitated with acid and removed from the solution. The nickel-free solution was reduced with hydrogen in an autoclave, producing cobalt powder, which was dried and briquetted. About 98 pct of the cobalt was recovered in the refining process. The overall cobalt recovery from nodule to cobalt powder was 89 pct.

Acid Sulfate Refining

The pregnant solution from the high-temperature, high-pressure H_2SO_4 leach process and the smelting and H_2SO_4 leach process were refined by the same method. First, the excess acid was neutralized with limestone to lower the H_2SO_4 content to 0.5 g/L. After filtering off the precipitated gypsum, the solution passed through solvent extraction with LIX to transfer the copper to the organic phase. The copper was stripped with spent copper

electrolyte containing 160 g/L H_2SO_4 . Then the copper was electrowon from the copper electrolyte. The copper raffinate was adjusted with ammonia to a pH of 4. During neutralization the tank was also aerated, causing the Fe, Mn, Mg, and Al impurities to precipitate. After centrifuging out the solids, solvent extraction collected the nickel in the organic phase. The nickel was stripped from the organic phase with spent nickel electrolyte. Then the nickel was electrowon from the nickel-rich electrolyte. The nickel raffinate was purified by the method described earlier for the LIX raffinate in the ammoniacal refining method. The final product was briquetted cobalt metal powder. Again, 89 pct of the cobalt was recovered from the nodules.

Acid Chloride Refining

The pregnant liquor was passed through a solvent extraction circuit to remove the Cu. The organic phase was stripped with spent copper electrolyte containing 160 g/L H_2SO_4 . Then the copper was electrowon from the copper-rich electrolyte. The pH of the raffinate from the copper extraction was adjusted to 4 with NaOH. The cobalt and some of the manganese were recovered by solvent extraction with TNOA. Both the cobalt and manganese were stripped from the organic and then the cobalt was precipitated with H_2S . This precipitate was treated by the same method reported earlier for the ammoniacal refining method, beginning with the pressure leaching of the mixed sulfide precipitates. The cobalt was recovered as a briquetted metal powder. About 99 pct of the cobalt was recovered from the nodules. The cobalt-free manganese solution went to the manganese recovery circuit. The cobalt-free raffinate was treated by solvent extraction to recover the nickel. The nickel was stripped from the organic phase with spent nickel electrolyte and recovered by electro-winning. The nickel-free raffinate was

combined with the cobalt-free raffinate and treated with H_2S to precipitate any residual Co, Ni, or Cu as sulfides. The remaining solution was evaporated, crystallizing manganese chloride salt. The salt was dried and fed to a high-temperature ($1,300^\circ C$) fused-salt electrolysis furnace where molten manganese metal was formed and periodically tapped. About 96, 99, and 94 pct of the Cu, Ni, and Mn were recovered from the nodules.

Research on manganese sea nodule processing is complete. The residues were environmentally acceptable and the process waste waters blended safely with seawater (9). At least five process options are available, but the economics are not favorable. At a price of \$17/lb, the calculated rate of return on investment was 4 to 6 pct. However, at least a 30-pct rate of return on investment is required to attract the necessary venture capital for sea nodule production.

MANGANESE SEA CRUSTS

Cobalt is also found in offshore manganese crust deposits located in the EEZ of Hawaii and Trust Territory of the Pacific Islands and affiliated territories. Cobalt-rich deposits on seamounts and

slopes have been estimated to contain up to 25 billion lb of Co. The average cobalt content of these crusts is 1.0 pct and they also contain 0.5 pct Ni, and 15 to 25 pct Mn (6). These deposits, 1-in thick layers, present a problem for selective mining. Equipment is being developed to break up the crust layer and leave most of the substrate behind, but this technology is still being tested. The mineralogy of the Pacific sea crusts is very similar to the Pacific nodules. Some places contain both crust and nodules. Even with a good selective mining system, beneficiation will be needed to separate the crust minerals from the substrate. Preliminary flotation tests have recovered 92 pct of the cobalt in a crust mineral concentrate. This research is not complete.

The extraction technology is expected to be identical to the sea nodule extraction methods. This resource still needs exploration, because very few samples have been taken from U.S. EEZ. The mining and beneficiation technology needs to be developed. The extraction technology is well developed, but the overall costs from mining through cobalt processing will be high.

DISCUSSION

The full potential for cobalt production from domestic sources is at least 19.4 million lb of Co per year. With the offshore manganese deposits, the amount doubles. A summary of each deposit's characteristics, beneficiation, extraction, and refining results is presented in table 1. Also, included is a six-level technological assessment of the research and development status for the beneficiation and cobalt processing of each ore.

The technology for Blackbird, Madison Mine, iron ore pyrite, laterites, and manganese sea nodules is nearly completely developed, but the economics are not favorable. Small technological improvements will have virtually no effect upon the economics of these operations. The most promising source of cobalt is the

spent copper leach solution. With all the mining and beneficiating costs paid by the primary copper production, cobalt can be extracted at a relatively low cost. The Missouri lead belt ores are also promising. Siegenite beneficiation from the copper concentrate is well developed, however the technology for extracting and refining the cobalt from the siegenite concentrate has not been fully explored. A secure domestic processing plant is needed to produce cobalt from the siegenite concentrate.

It appears that beneficiation can not increase the bulk sulfide grade from the lead mill tailings without losing recovery. Because of the dolomite gangue, cobalt processing technology for this concentrate will probably be limited to an ammoniacal leach. The

applicability of this technology needs to be established so that the technical feasibility and economics of cobalt recovery from this source can be evaluated.

The pyrite from some primary copper operations also shows potential for significant cobalt recovery. The technology for beneficiation and cobalt processing still needs research, but without the mining cost burden, this cobalt source should be one step closer to favorable economic production of cobalt.

Both the Duluth Gabbro and Yakobi Island deposits show potential for cobalt. The beneficiation technology for the Duluth Gabbro deposit is complete, but the cobalt processing technology needs to be established on the Co-Ni concentrate. Cobalt production from this deposit appears to be many years into the future. For each pound of Co produced from this deposit, 95 lb of Cu and 14 lb of Ni are also produced. The present market conditions for copper offer no incentive for exploiting this deposit for copper.

The deposit on Yakobi Island is in a similar predicament. The beneficiation and processing technologies need to be explored. With 6 lb of Cu and 10 lb of Ni produced for each pound of cobalt, there is also little hope for development in the near future.

Economic evaluation of cobalt deposits should help to focus research on the deposits with the best future for economical production. A Bureau cobalt availability study on cobalt from world resources indicated that the mining and beneficiation costs are a substantial portion of the operating costs (20). That is why byproduct recovery from present copper, lead, and zinc operations, where the mining and beneficiation costs are already paid, have the highest potential for immediate cobalt production. Over 5 million lb of Co per year could be produced from these operations. Research on these sources should be given the highest priority. Even with virtually unlimited offshore cobalt resources, the high mining and ore transportation costs associated with the offshore mining will postpone the development of these resources for many years (14).

Under the present economic conditions cobalt could be made available within 2 to 3 yr only as a byproduct from present operations. With higher but stable commodity prices, many of the other cobalt resources could be available in 4 to 5 yr. In the event of a national emergency, the present cobalt stockpile could meet U.S. cobalt demand for 3 yr (4) or with restrictions the stockpile could meet essential U.S. needs for up to 6 yr (22).

TABLE 1. - U.S. cobalt resource summary

Resource	Total Co, million lb	Co grade, pct	Planned annual Co production, million lb	Byproduct metal production			Beneficiation			Process technology	
				Cu-Co	Ni-Co	Mn-Co	Co grade, pct	Co rec, pct	Tech assess- ment ¹	Co rec, pct	Tech assess- ment ²
Blackbird.....	60	0.65	4	2.7	0.02	Nap	5	80	4	75	4
Duluth Gabbro.....	184	.025	5	95	14	Nap	.23	32	4	Nap	1
Yakobi Island.....	14	.04	NA	6	10	Nap	Nap	Nap	0	Nap	0
Madison Mine.....	21	.17	1.5	NA	1	Nap	1.5	95	5	92	5
Missouri lead:											
Cu conc.....	80	.015	.6	7	1	Nap	2.8	20	4	19	3
Pb tailings.....	80	.015	1.3	9	1	Nap	.11	33	2	Nap	0
Cu pyrite conc.....	>25	.009	>1.0	16	Nap	Nap	.06	30	1	Nap	0
Spent Cu leach solution.	>50	.003	>1.5	2.5	1	Nap	Nap	Nap	Nap	96	3
Fe pyrite conc.....	56	.04	1.5	NA	Nap	Nap	1.0	NA	5	NA	5
Laterites.....	96	.08	3	.1	12.5	4	Nap	Nap	Nap	67	4
Sea nodules.....	>1,000	.26	15	2	4	100	Nap	Nap	Nap	89	4
Sea crusts.....	>25,000	.66	NA	.1	1	25	.25	90	1	80	3

NA Not available.

Nap Not applicable.

¹For beneficiation:

- 0 - Exploration continuing
- 1 - Exploration, mineralogy, and size of deposit defined
- 2 - Liberation and exploratory beneficiation complete
- 3 - Laboratory process developed
- 4 - Pilot plant testwork completed
- 5 - Commercial production

²For process technology (extraction and refining):

- 0 - Laboratory testing continuing
- 1 - Laboratory process defined
- 2 - Continuous testing completed
- 3 - Commercial unit operations available
- 4 - Pilot plant testing completed
- 5 - Commercial production

CONCLUSIONS

Byproduct recovery of cobalt from present copper, lead, and zinc operations has the highest potential for immediate cobalt production. This includes spent copper leach solutions from primary copper production, the siegenite concentrate from Missouri lead-zinc-copper production, and pyrite concentrates from some primary copper mines.

Technology research for the Blackbird, Madison Mine, iron ore pyrite, laterites, and manganese sea nodules is nearly complete. Development depends upon favorable economic conditions. Additional research on these resources should be limited to approaches that promise to cut the total processing costs by over 50 pct.

Research on the Duluth Gabbro and Yakobi Island deposits is not complete

and they will not be developed until favorable copper and nickel markets reappear. This will be many years in the future, so that research is very long range.

Except for beneficiation of the sea crusts, research on sea nodules and sea crusts is complete. Development of these resources is many years off due to economic, legal, and political problems.

Solution mining or heap leaching should be explored for the lead mill tailings, laterites, and Duluth Gabbro deposits as an alternative lower cost mining and extraction process. Once the cobalt is in the solution, the Bureau's spent copper leach solution refining technology could be used to recover the cobalt.

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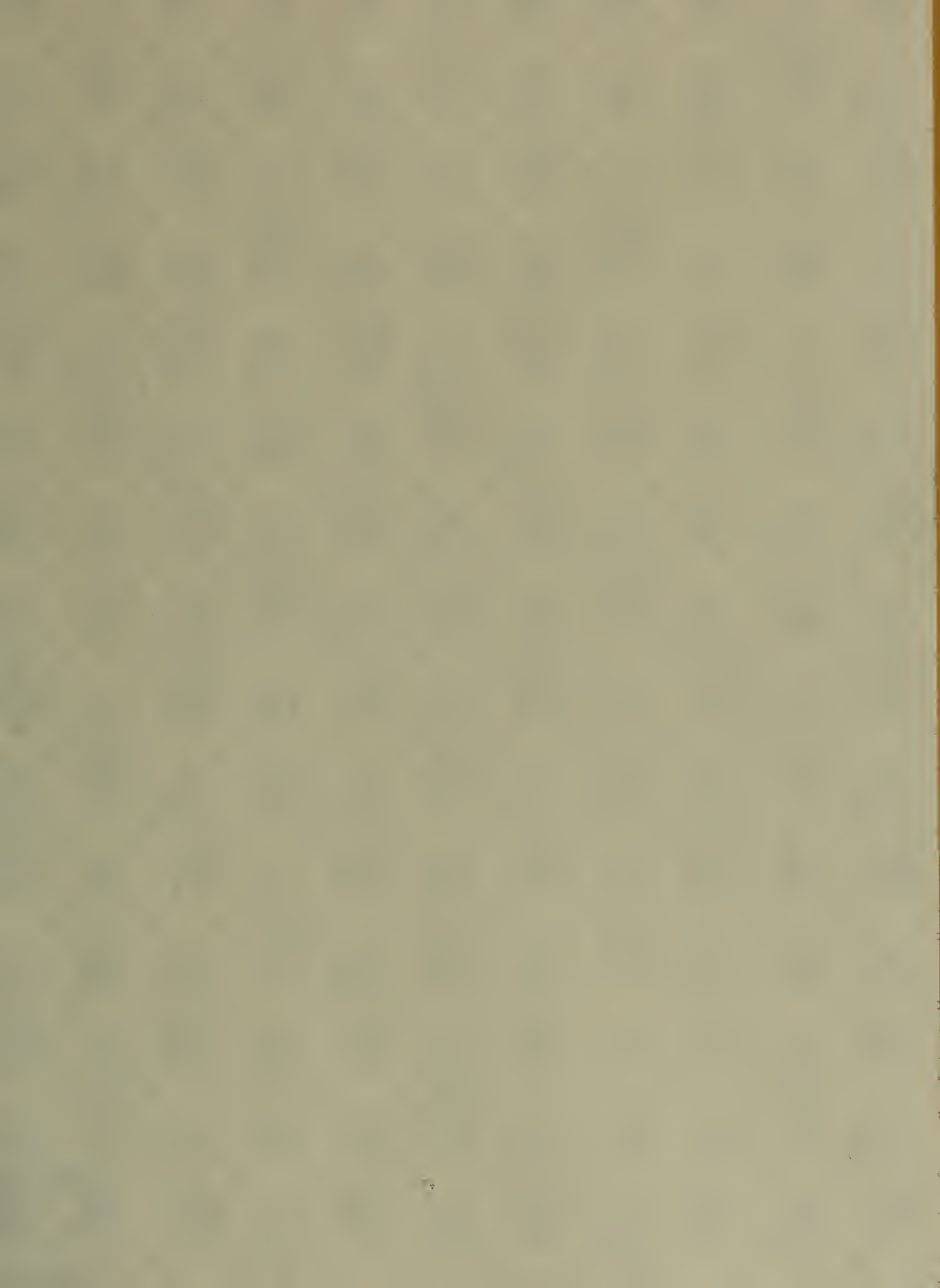


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